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AN APPARATUS FOR DIFFERENTIAL-THERMAL ANALYSIS OR ISOTHERMAL DECOMPOSITION WITH SIMULTANEOUS MASS-SPECTROMETRIC ANALYSIS

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AN APPARATUS FOR DIFFERENTIAL-THERMAL ANALYSIS OR ISOTHERMAL DECOMPOSITION WITH SIMULTANEOUS MASS-SPECTROMETRIC ANALYSIS

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ABSTRACT. A description of a very simple system allowing the performance of analyses of the products of thermolysis in parallel with a study of the kinetics of isothermal decomposition or differential-thermal analysis of compounds. The device involves recording of the pressure of gases liberated with simultaneous sampling of gaseous products of decomposition for mass-spectrometric analysis.

The literature contains descriptions of a number of installations and methods for the performance of isothermal decomposition or differential-thermal analysis of solid compounds with simultaneous mass spectrometric analysis of the gaseous products of decomposition [1-3]. In the variants described in the literature, however, the performance of this analysis is frequently hindered by methodological complexities (for example, the necessity of placing the sample and heating system into an ion source or of assuring strictly constant flow of the carrier gas for the products and choking the gas).

We have produced and will describe below an extremely simple system allowing the performance of analysis of the products of thermolysis in parallel with a study of the kinetics of isothermal decomposition or differential-thermal analysis of compounds.

The reaction vessel, which is at the same time a part of the inlet system, is shown schematically on the figure.

Decomposition of the preparate in the differential-thermal analysis mode or during isothermal heating is controlled to an accuracy of 0.1 mm Hg pressure of the liberated gases by the Bourdon manometer, used here as a null-instrument, operating in conjunction with a high accuracy MChR-2 mercury manometer.

Preliminary evacuation of the vessel (to a pressure of $\approx 10^{-3}$ mm Hg) is performed with valve (7) open, using the evacuation system of the MKh-1302 mass spectrometer.

Thermostatting of the vessel or linear variation of the temperature (in the differential-thermal analysis mode) is achieved by massive furnace (13).

The operating mode of the furnace is controlled by a thermostatting system using the regulating potentiometer of the EPD-12 (17) or is created on

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[1] Numbers in the margin indicate pagination in the foreign text.

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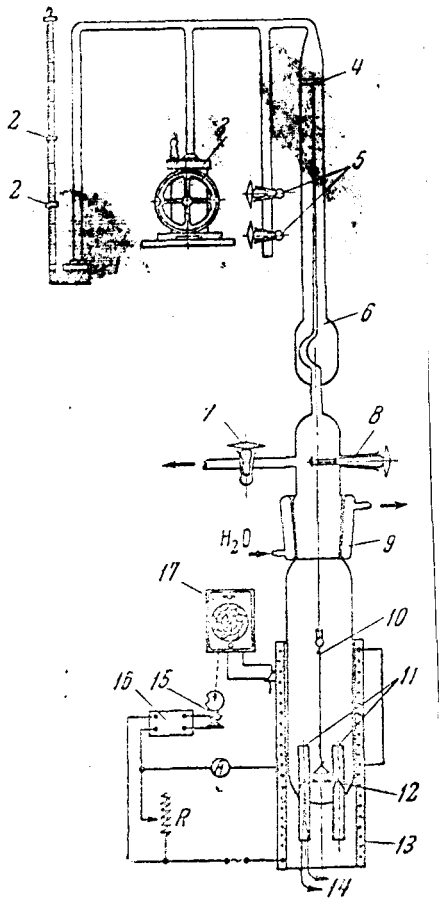

Diagram of Installation: 1, 2, Mercury manometer type MChR-3; 3, Komovskiy pump; 4, Scale of Bourdon manometer; 5, Stopcocks; 6, Bourdon manometer; 7, Valve connecting vessel with type MKh-1302 ampule system; 8, Valve for introducing charge; 9, Cooled jacket; 10, Support; 11, Vessels for analysis; 12, Glass dish with charge; 13, Furnace; 14, Thermocouple; 15, Positional regulator of type EPD potentiometer; 16, Type TRR relay; 17, Type EPD-12 potentiometer.

the basis of the linear temperature change system of a Kurnakov pyrometer.

When isothermal decomposition of a charge is being performed, the material is placed in container (12), placed through valve (8) into the preheated portion of the reaction vessel. /2399

When the unit operates in the differential-thermal mode, the preparate to be investigated and the standard sample are placed in containers soldered to the bottom of the vessel, the thermocouples in this case being separated from the charge by the thin glass bottoms of the containers.

Gas samples for analysis are taken through stopcocks (7) by filling one of the ampules connected to the inlet system of the MKh-1302 mass spectrometer [4] with the gas to be analyzed. Due to the small volume of the samples taken, this operation does not interfere with the study of the kinetics of the thermolysis.

The unit which we have described has been operated in both modes, and has been found to be quite simple to assemble and operate, as well as sufficiently reliable and sensitive in the differential-thermal analysis mode.

Conclusions

An installation is described for the study of the kinetics of thermal decomposition of solid materials and performance of differential-thermal analysis, featuring recording of pressures of gases liberated with simultaneous sampling of the gaseous products of decomposition for mass-spectrometric analysis on the MKh-1302 mass spectrometer.

The work was performed at Tomsk Polytechnical Institute in the Chair of Radiation Chemistry.

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